

PRODUCTION OF PDMS/PEG-PVDF THIN FILM COMPOSITE MEMBRANE FOR  
CO<sub>2</sub>/N<sub>2</sub> SEPARATION

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## ABSTRACT

A thin film composite (TFC) membrane was prepared for separation of carbon dioxide ( $\text{CO}_2$ ) and nitrogen ( $\text{N}_2$ ). The support layer was fabricated from different concentrations of polyethylene glycol (PEG) and the coating layer was prepared from different concentration of polydimethylsiloxane (PDMS). The concentration ratio of PDMS to PEG is 2:1, 1:1 and 1:2. Permeances and selectivity of the prepared membranes  $\text{CO}_2$  and  $\text{N}_2$  gases and their mixtures were measured under trans membrane pressure of 1 bar. The different concentration ratio of PDMS to PEG plays an important role for permeability and selectivity of  $\text{CO}_2/\text{N}_2$  gas separation. It was shown that the highest permeability and selectivity was belong to membrane coating ratio of 1:2 (PDMS: PEG) followed by 2:1 (PDMS: PEG) and lastly 1:1 (PDMS:PEG). The result of permeation test was supported by the results from SEM and FTIR. SEM showed the membrane morphology for surface and cross section membrane. While, FTIR showed the functional group that exists within molecules in membranes.

## ABSTRAK

Membran komposit filem nipis yang telah disediakan untuk pemisahan karbon dioksida ( $\text{CO}_2$ ) dan nitrogen ( $\text{N}_2$ ). Lapisan sokongan direka dari kepekatan yang berbeza polietilena glikol (PEG) dan lapisan salutan telah disediakan dari kepekatan yang berbeza polydimethylsiloxane (PDMS). Nisbah kepekatan PDMS untuk PEG 02:01, 01:01 dan 01:02. Kebolehtelapan dan pemilihan  $\text{CO}_2$  membran yang disediakan dan gas  $\text{N}_2$  dan campuran ini diukur di bawah tekanan membran trans 1 bar. Nisbah kepekatan yang berlainan PDMS untuk PEG memainkan peranan yang penting untuk kebolehtelapan dan pemilihan bagi pemisahan  $\text{CO}_2/\text{N}_2$  gas pemisahan. Ia menunjukkan bahawa kebolehtelapan tertinggi dan pemilihan adalah milik membran salutan nisbah 1:2 (PDMS: PEG) diikuti oleh 02:01 (PDMS: PEG) dan akhir sekali 1:1 (PDMS: PEG). Hasil ujian penyerapan disokong oleh keputusan dari SEM dan FTIR. SEM menunjukkan morfologi membran untuk permukaan dan keratan rentas membran. Sementara itu, FTIR menunjukkan kumpulan berfungsi yang wujud dalam molekul dalam membran.

The storage for CO<sub>2</sub> has been identified as one potential solution to greenhouse gases driven climate change. Membrane have been investigated for over 150 years and since 1980 gas separation membrane have been used commercially (Powell, 2005). Gas membrane separation is one of the method uses in industry. The advantages of using gas membrane separation are energy efficiencies and simplicity of membrane gas separation makes it extremely attractive for CO<sub>2</sub> capture.

## **1.2 Problem Statement**

Gas membrane separation is one of the method uses to capture and storage CO<sub>2</sub>. Instead of using solvent absorption, gas membrane separation is more compact, energy efficient and possibly more economical. The highest permeability and selectivity of membrane depends on material use for coating. Furthermore, the chosen of materials for coating plays an important role to get highest selectivity and permeability of CO<sub>2</sub>/N<sub>2</sub> gas separation. The different concentration of coating material also plays an important role because the higher concentration of coating material, the higher will be their selectivity and permeability for CO<sub>2</sub>/N<sub>2</sub> gas separation.

Uses membrane have been rapidly growing in the application of gas separation process. This research is concentrated on the ability performance of the membrane to separate the CO<sub>2</sub> and N<sub>2</sub>. As mention before, CO<sub>2</sub> is a gas that can contribute to global warming and need to reduce CO<sub>2</sub> emission.

### **1.3 Research Objectives**

Based on problem statement described in the previous section, therefore the objective of this research are:

- To produce TFC membrane.
- To characterise selectivity of TFC membrane.
- To investigate the permeability and selectivity of TFC membrane.

### **1.4 Scope of Research**

In order to achieve the above mentioned objective, the following scope has been drawn:

- Develop a best formulation solution and produce the TFC membrane by using PDMS, PEG as coating layer and PVDF membrane as support layer.
- Study on performance of TFC membrane.
- Characterize on TFC membrane physically and chemically by using FTIR and SEM.

## **1.5 Rational and Significance of Research**

- a) To increase the permeability and selectivity of CO<sub>2</sub> gas separation by membrane system.
- b) To developed the economical process for CO<sub>2</sub> capture to sustain supply the increasing gas demand.
- c) Build up extremely versatile capable medium to produce porous membrane for all separation stage.

## **CHAPTER 2**

### **LITERATURE REVIEW**

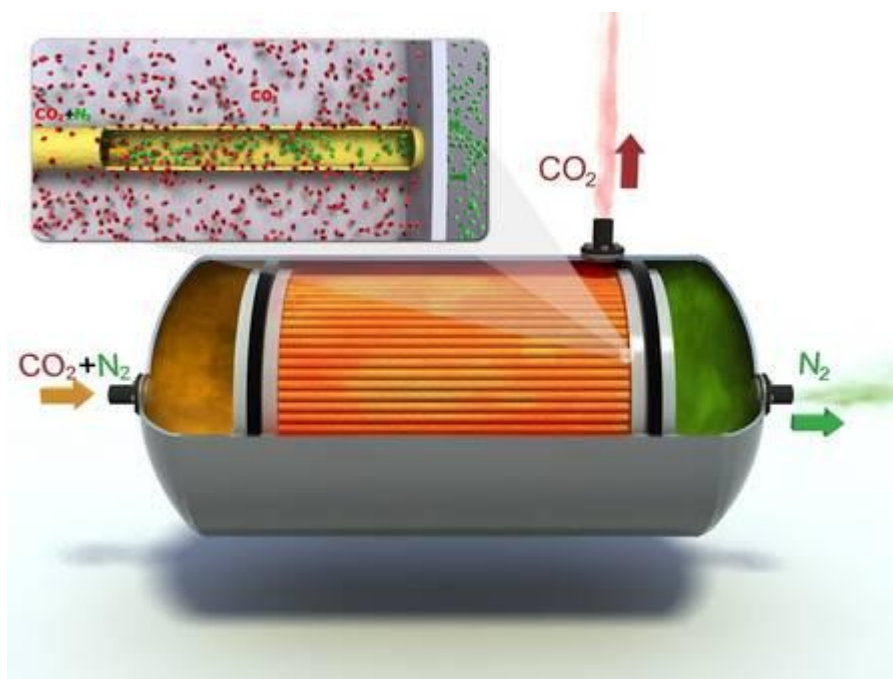
#### **2.1 Introduction**

##### **2.1.1 Membrane**

Nowadays, membrane technology for gas separation are widely use because gas separation offer overall advantages such as reducing environmental impact and cost of industrial process. Based on Freeman (2005), gas separation membranes offer a number of benefits over other gas separation technologies. One of the benefit is membrane gas separation does not required a phase change. In addition, gas separation membrane unit are smaller than other types of plants, like stripping plants and therefore have relatively small footprints.

The term membrane most commonly refers as thin, film-like structure that separate or restrict certain molecules to flow through membrane. A membrane can be defined essentially as a semi-permeable barrier, which separates a fluid and restricts transport of various chemicals in a selective manner. A membrane can be homogenous or heterogeneous, symmetric or asymmetric in structure, solid or liquid

can carry a positive or negative charge or be neutral or bipolar. The concept of membrane is simple and similar to a filter. Generally, CO<sub>2</sub> capture membranes are designed to be selective for small gases for example allowing N<sub>2</sub> to pass through while leaving a pure stream of CO<sub>2</sub> behind. **Figure 2.1** shows the separation of carbon dioxide and nitrogen used in industrial today.



**Figure 2.1** Separation of Carbon Dioxide and Nitrogen



### 2.1.2 Types of Membrane

The efficiency of the gas separation is based on permeability and selectivity of membrane material. Various types of membrane are widely use and produce today such as micro porous membranes, homogeneous membrane, asymmetric membrane, nonporous and dense membrane. **Table 2.1** show the types of membrane and their characteristic.

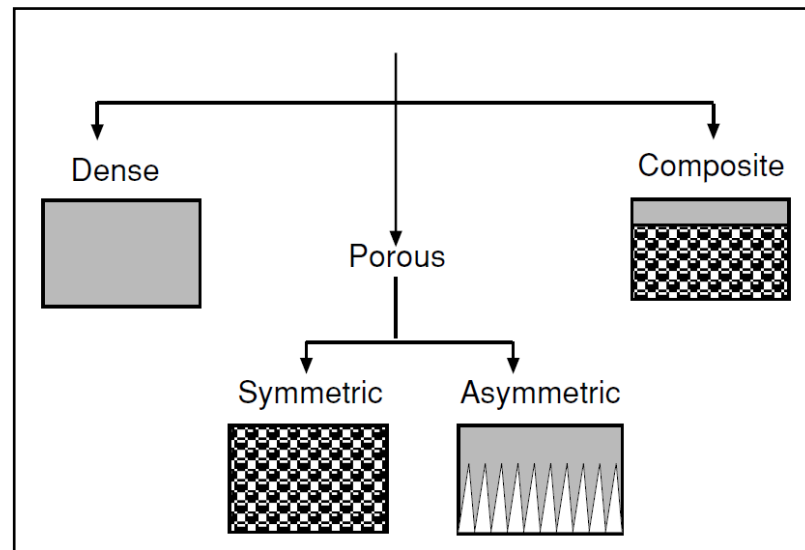
**Table 2.1:** Types of Membrane and Their Characteristic

Types of membrane	Characteristics	References
Micro porous membranes	The membrane behaves almost like a fibre filter and separates by a sieving mechanism determined by the pore diameter and particle size. Material that are used in making such membrane are ceramics, graphite, metal oxides, polymers and etc. The pores in the membrane may vary between 1 nm-20 microns.	Srikanth, (2004)
Homogeneous membranes	This is a dense film through which a mixture of molecules is transported by pressure, concentration or electrical potential gradient. By using these membranes, chemical species of similar size and diffusivity can be separated efficiently when their concentrations differ significantly.	Srikanth, (2004)
Asymmetric membranes	An asymmetric membrane comprises a very thin (0.1-1.0 micron) skin layer on a highly porous (100-200 microns) thick substructure. The thin skin acts as the selective membrane. Its separation	Srikanth, (2004)

	characteristics are determined by the nature of membrane material or pore size, and the mass transport rate is determined mainly by the skin thickness. Porous sub-layer acts as a support for the thin, fragile skin and has little effect on the separation characteristics.	
Electrically charge membranes	These are necessarily ion-exchange membranes consisting of highly swollen gels carrying fixed positive or negative charges. These are mainly used in the electro dialysis. Electrically charged membranes can be dense or micro porous, but are most commonly very finely micro porous, with the pore walls carrying fixed positively or negatively charged ions. A membrane with fixed positively charged ions is referred to as an anion-exchange membrane because it binds anions in the surrounding fluid. Similarly, a membrane containing fixed negatively charged ions is called a cation-exchange membrane. Separation with charged membranes is achieved mainly by exclusion of ions of the same charge as the fixed ions of the membrane structure, and to a much lesser extent by the pore size. The separation is affected by the charge and concentration of the ions in solution.	Srikanth, (2004)
Flat film membranes	Materials for flat film membranes are usually designs employ textiles, non-woven fabrics or porous polymeric sheets. Composite membranes of this construction enable the separating layer thickness to be reduced to a few microns. When high flux polymers are employed, the transport resistance of the non-selective support layer can	Nunes, (2001)

	become a significant resistance. The effect of resistances in series with the separating layer resistance has the effect of reducing the overall membrane selectivity. Hence, much attention is given to minimizing the supporting layer's resistance.	
Nonporous, Dense Membranes	Nonporous, dense membranes consist of a dense film through which permeants are transported by diffusion under the driving force of a pressure, concentration, or electrical potential gradient. The separation of various components of a mixture is related directly to their relative transport rate within the membrane, which is determined by their diffusivity and solubility in the membrane material. Thus, nonporous, dense membranes can separate permeants of similar size if their concentration in the membrane material (that is, their solubility) differs significantly. Most gas separation, pervaporation, and reverse osmosis membranes use dense membranes to perform the separation.	

Membrane can be classified into few types. The classification of membrane can help to improve the membrane application by knowing to the membrane morphology. Membranes can be classified, according to their morphology. Before this, types of membrane have been shown. Now, **Figure 2.2** shows the membrane classification according to the morphology.



**Figure 2.2** Membrane Classifications According to the Morphology (Nunes, 2006)

### 2.1.3 Mechanism for Gas Separation

There are two main membrane permeation mechanisms for gas separation mechanism such as dense membranes and porous membranes. Usually, dense membranes have high selectivity and gives low fluxes. However, larger pore gives higher fluxes but decrease selectivity.

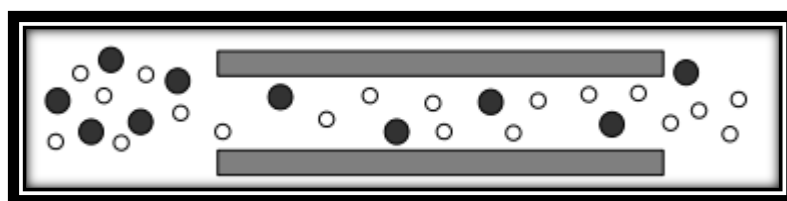
#### 2.1.3.1 Dense Membrane Separation Mechanism

The solution or diffusion mechanism is the most commonly used physical model to describe gas transport through dense membrane. A gas molecule is

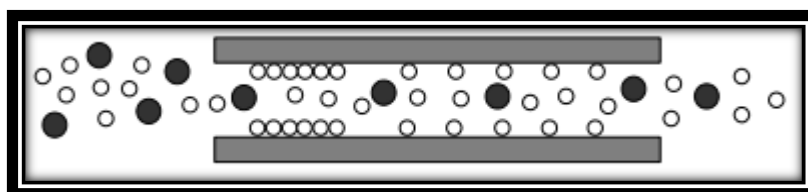
adsorbed on one side of the membrane, dissolves in the membrane materials, diffuses through the membrane and desorbs on the other side of the membrane.

### 2.1.3.2 Porous Membrane Separation Mechanism

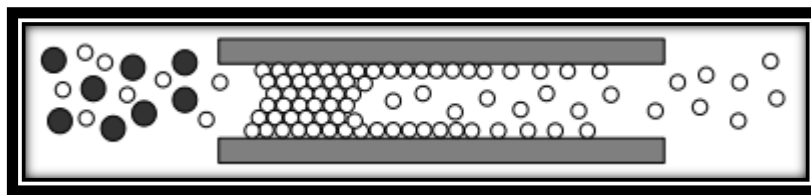
There are four types of diffusion mechanisms can be utilized to effect separation in porous membranes. In some cases, molecules can move through the membrane by more than one mechanism. These mechanisms can be described by Knudsen diffusion. The Knudsen diffusion gives low separation selectivity compare with surface diffusion and capillary condensation. In order to get high selectivity, shape selective separation and molecular sieving plays important role. The parameter need to be considered for separation mechanism depends strongly on pore size distribution, temperature, pressure and interactions between gases being separated and the membrane surfaces. **Figure 2.3** shows the transport mechanisms in porous membranes by Knudsen diffusion, surface diffusion, capillary condensation, molecular sieving.



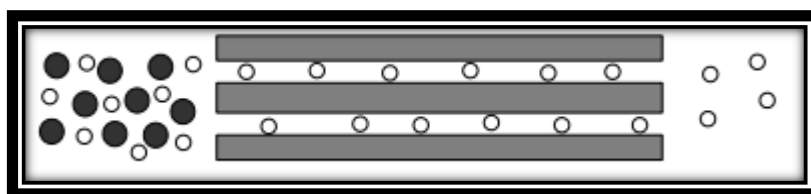
(i) Knudsen Diffusion



(ii) Surface Diffusion



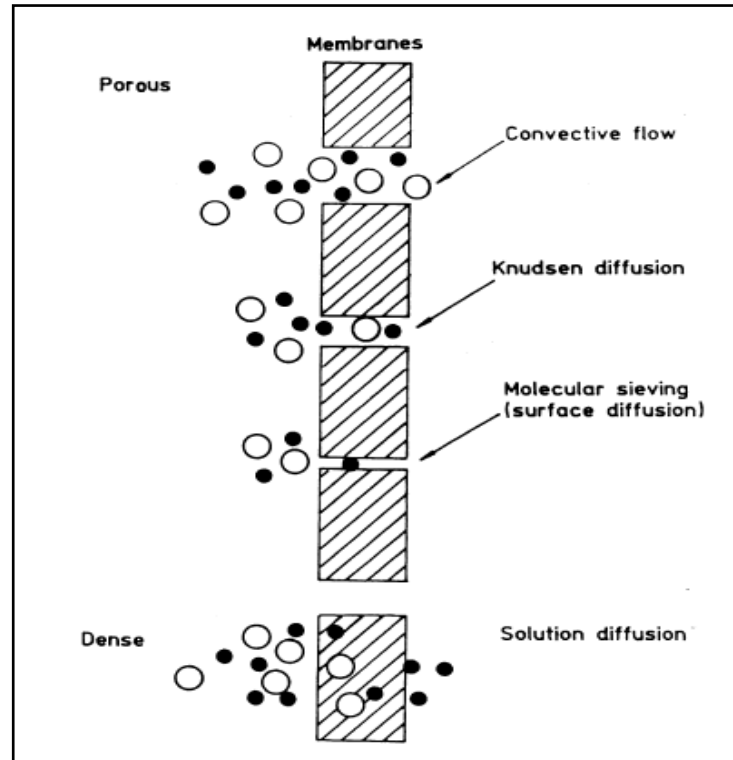
(iii) Capillary Condensation



(iv) Molecular Sieving

**Figure 2.3** Transport mechanisms in porous membranes: (i), (ii), (iii), (iv); (S.C.A. Kluiters, 2004)

Various mechanisms for gas transport across membranes have been proposed depending on the properties of both the permeant and the membrane. These include Knudsen diffusion, the molecular sieve effect and solution diffusion mechanism. **Figure 2.4** show the schematic presentation of mechanisms for permeation of gases through membrane.



**Figure 2.4** Schematic presentation of mechanisms for permeation of gases through membrane (Pandey, 1999)

## 2.2 Membrane Process

Membrane processing is a technique that permits concentration and separation without the use of heat. Particles are separated on the basis of their molecular size and shape with the use of pressure and specially designed semi-permeable membranes.

**Table 2.2** show the application of membrane separation and membrane type. During the past two decades membrane separation processes have been developed and optimized for even large scale industrial applications. The most important of the processes include:

- Microfiltration and ultra-filtration
- Reverse osmosis
- Electro dialysis
- Gas separation
- Evaporation

**Table 2.2:** Application of Membrane Separation and Membrane Type

<b>Membrane process</b>	<b>Membrane type</b>	<b>Application</b>
Reverse osmosis	Asymmetric membrane	<ul style="list-style-type: none"> <li>• used on a zinc sulfate rinse</li> <li>• operating on various copper sulfate rinses</li> <li>• Cadmium and chromium rinsewaters are also treated with RO</li> <li>• desalination and demineralization of saline water</li> <li>• demineralized or potable water</li> </ul>
Ultrafiltration	Asymmetric membrane	<ul style="list-style-type: none"> <li>• Biological buffers</li> <li>• Protein chemistry</li> <li>• Blotting and hybridization</li> </ul>
Microfiltration	Symmetric membrane	<ul style="list-style-type: none"> <li>• Purification of fluids in semiconductors</li> <li>• sterilization (in pharmaceutical)</li> </ul>



Pervaporization	Non-porous membrane, composite membrane	<ul style="list-style-type: none"> <li>• separation of more polar organics from less polar ones,</li> <li>• especially when water is absent</li> <li>• -water removal from liquids organics</li> <li>• -organic separation</li> <li>• -separation of liquid mixtures, especially of aqueous-organic azeotropes</li> </ul>
Gas permeation	Non-porous membrane, asymmetric composite	<ul style="list-style-type: none"> <li>• Prepurification of natural gas</li> <li>• hydrogen separation</li> <li>• biogas processing</li> <li>• splitting gas streams</li> <li>• removal or recovery of specific gases</li> </ul>
Electro dialysis		<ul style="list-style-type: none"> <li>• separation of microsolutes and salts from macromolecular solution.</li> <li>• Removal of dissolved ions</li> </ul>

### 2.3 Membrane Application

The applications of gas membranes separation are listed as below. **Table 2.3** shows a gas separation and its application (Nunes, 2006).

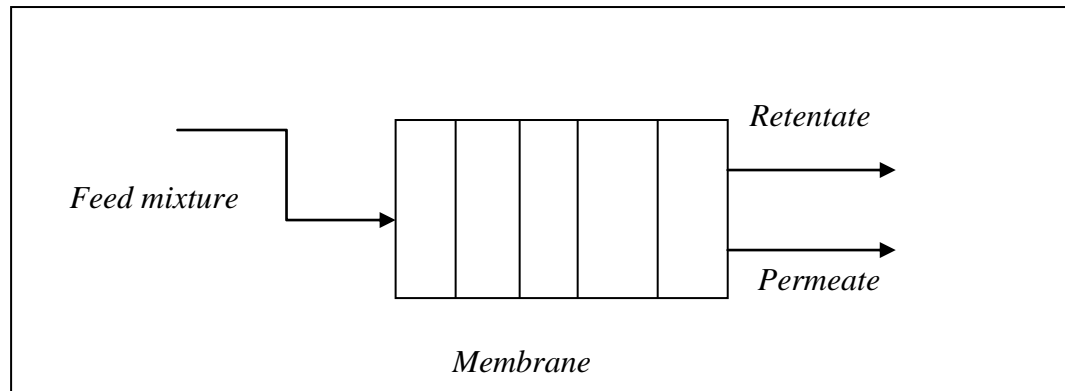
**Table 2.3:** Gas Separation and Its Applications (Nunes, 2006)

Gas Separation	Application
H <sub>2</sub> /N <sub>2</sub>	Syngas ratio adjustment
CO <sub>2</sub> /Hydrocarbon	Acid gas treatment and landfill gas upgrade
O <sub>2</sub> /N <sub>2</sub>	Nitrogen generation and oxygen enrichment
H <sub>2</sub> /CO	Syngas ratio adjustment
H <sub>2</sub> S/Hydrocarbon	Sour gas treating
H <sub>2</sub> O/hydrocarbon	Natural gas dehydration
H <sub>2</sub> /Hydrocarbon	Refinery hydrogen, recovery
H <sub>2</sub> O/air	Air dehydration
Hydrocarbons/air	Pollution control, hydrocarbon recovery
Hydrocarbons from process streams	Organic solvent recovery, monomer recovery.

## 2.4 Membrane in Gas Separation

The process that is considered in this work is membrane based gas separation process using polymeric membrane and organic membrane. **Figure 2.5** shows general membrane process that show how separation flows in membrane. As mention before, a membrane act as a barrier that separate into retentate, permeate and restrict transport of various chemical species in a selective manner. The stream that permeates through the membrane is the permeate stream, while the one retained by the membrane is the retentate. There are two characteristic of dictate the membrane performance, permeability and selectivity. Permeability is the flux of a specific gas

through the membrane and selectivity is the membrane's preference to pass one gas species and not another (Collin, 2008).



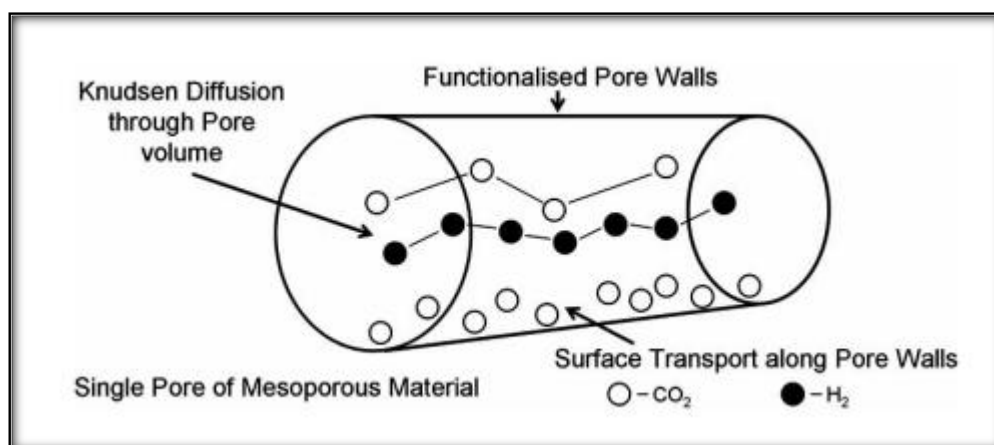
**Figure 2.5** General Membrane Process

#### 2.4.1 Polymeric Membrane

Polymeric membrane is one of the types of membrane used for gas separation. Polymeric membranes are the most popular membranes because of their high performance, easy synthesis, long life, good thermal stability, adequate mechanical strength and high resistance to gases and chemicals (Sadrzadeh, 2009). Moreover, polymeric membranes are characterized as a thin, dense selective surface skin on a less dense porous support that is non-selective (Colin, 2008). Membrane can be classified into two classes. There are porous and non-porous. A porous membrane is a rigid, highly voided structure with randomly distributed inner-connected pores. Non-porous that can be known as dense membranes provide high selectivity or separation of gases from their mixtures but the rates transports of the gases are usually low.

### 2.4.2 Inorganic Membrane

Inorganic membrane was representing as an alternative gas separation technology. There are two types of inorganic membrane, porous and non-porous. Non-porous membranes are generally used in highly selective separation of hydrogen ( $H_2$ ), where transportation is through alloy of palladium. While, porous inorganic membranes are generally cheaper but less selective (Colin, 2008). The large size  $CO_2$  compare with  $H_2$ , can result in achieving  $CO_2$  rich permeate stream by simple molecular sieving. However, inorganic membrane overcomes this by functionalizing the pores of the membrane to increase the  $CO_2$  loading. **Figure 2.6** shows schematic of inorganic membrane operation through Knudsen and surface diffusion (Colin, 2008).



**Figure 2.6** Schematic of Inorganic Membrane Operation through Knudsen and Surface Diffusion (Colin, 2008)

In order to get high selectivity and permeability, the correct membrane material and correct processing condition are needed. Selectivity is the ratio of the permeability of the  $CO_2$  to other component in the stream (Mutun, 2010). The higher

the permeability, the less membrane is required for a given separation and result in lower cost. The higher the selectivity, the lower will be the losses of hydrocarbons when CO<sub>2</sub> is removed. **Table 2.4** shows the materials for gas separation membrane.

**Table 2.4:** Materials for Gas Separation Membrane (Nunes, 2001)

Organic polymer	Inorganic materials
Polysulfone, polyethersulfone	
Celluloseacetate	Carbon molecular sieves
Polyimide, polyetherimide	Nonporous carbon
Polycarbonate (brominated)	Zeolites
Polyphenyleneoxide	Ultramicroporous amorphous silica
Polymethylpentene	Palladium allys
Polydimethylsiloxane	Mixed conducting perovskites
Polyvinyltrimethylsilane	

## 2.5 Membrane Modification

Based on Jiang et al., (2008) research, rubbery thin film composite membrane have been used in petroleum and chemical industries to preferentially permeate volatile organic compound for many decades with the benefit of saving energy and

valuable hydrocarbons. Poly(dimethylsiloxane) (PDMS) is a favourable coating material in polymeric membrane-based gas separation due to its hydrophobicity and thermal stability. Its low glass transition temperature ( $-129\text{ }^{\circ}\text{C}$ ) results in extremely high permeation through the membrane for a wide range of gases due to the flexible polymer backbone. However, it is limited in mechanical strength and selectivity particularly for highly condensable hydrocarbons in admixture with permanent gases. For example, Jiang, (2008) showed that PDMS coated membrane can be plasticized by propane and propylene, resulting in significant permeance increases for both hydrocarbons and nitrogen in a quaternary mixture of propane, propylene, ethylene and nitrogen, therefore, the selectivity of hydrocarbons to nitrogen were not as high as expected.

The materials used to fabricate membranes can be categorized as organic and inorganic. Organic materials are either cellulose-based or consist of modified organic polymers. Inorganic materials such as metals and ceramics are used in niche industrial applications, but are often cost prohibitive in wastewater treatment. Based on Stephenson, (2000) a partial list of available materials is given below:

- Titanium Dioxide/Zirconium
- Dioxide Cellulose Acetate
- Polyether Sulfone(PES)
- Poly Acrilo Nitrile (PAN)
- Polyamide, Polyimide
- Polyethylene (PE)
- Polypropylene (PP)
- Poly Tetra Fluoro Ethylene (PTFE)
- Poly Vinylidene Fluoride (PVDF)
- Poly Vinyl Chloride (PVC)

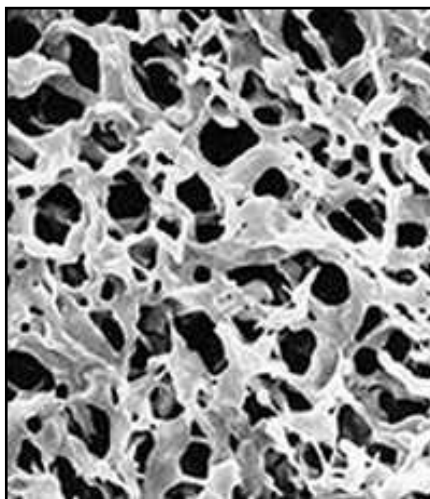
### 2.5.1 Polyvinylidene Fluoride (PVDF) Membrane

The use of polyvinylidene fluoride (PVDF) membrane as a support substrate layer for making a polyamide thin-film composite (TFC) has been hindered by the hydrophobic nature of PVDF. By the research of Kim et al., (2009) the objective of their study was to investigate whether the PVDF based microfiltration membrane could be modified to make it suitable for polyamide TFC membrane preparation. From their research, plasma modification of PVDF membranes enhances oxygen/methane (1:1) gas mixture is effective.

Nowadays, the types of support polysulfone (PSF) or polyethersulfone (PES) are mostly used as an interfacial polymerization. Due to the A.L. Ahmad (2008) and group, they found that Polypropylene (PP) and polyvinylidenefluoride(PVDF) are very attractive materials as a hydrophobic support for forming a TFC membrane due to its high durability and resistance to chemicals, pH variations, and a substantially wide range of solvents. They found that, the TFC with 6% w/v of glutaraldehyde based on PVDF achieved the highest permeance of 881.70 GPU and 18.08 for selectivity through the increase in effective layer and skin layer thickness. This TFC promises to provide porous and hydrophobic membranes for use in membrane gas absorption (MGA) processes. The absorption of CO<sub>2</sub> in deionized water was studied in MGA system in which the mass transfer coefficient (K) and CO<sub>2</sub> flux decreased with increasing CO<sub>2</sub> concentration in feed stream.

Polymeric membranes can be prepared by phase inversion process. The remixing of a polymer solution can be induced by compositional changes of constituents due to mass transfer in a system. Based on the research of Yuan et al., (2008) they are produced asymmetric blend hollow fibre membrane using new casting dope containing poly(vinylidene fluoride) (PVDF)/Thermoplastic polyurethane (TPU)/Polyvinylpyrrolidone (PVP) N, N dimethylacetamide(DMAc).

The additives are widely used for structure control of membranes such as polyvinylpyrrolidone(PVP) and Polyethylene glycol (PEG). **Figure 2.7** show the PVDF membrane.



**Figure 2.7** Polyvinylidene fluoride (PVDF) Membrane

### 2.5.2 Polyethylene Glycol (PEG)

Based on Wongchitphimon *et al.*, (2010), Polyethylene glycol (PEG) is one of the additives used to promote pore formation in the polymeric membranes. PEG is a linear polyether compound available in a variety of molecular weight, which is indicated by a numeric suffix followed the abbreviation (PEG). Its general formula is expressed as  $\text{H}(\text{OCH}_2\text{CH}_2)_n\text{OH}$ , where  $n$  is the average number of repeating oxyethylene groups. PEG is water-soluble. It is also soluble in many organic solvents including aromatic hydrocarbons. Thus PEG has been reported as a pore former to enhance the permeation properties for not only hydrophilic membranes but also hydrophobic membrane preparation, (Wongchitphimon *et al.*, 2010).



### 2.5.3 Poly (Dimethyl Siloxane) PDMS

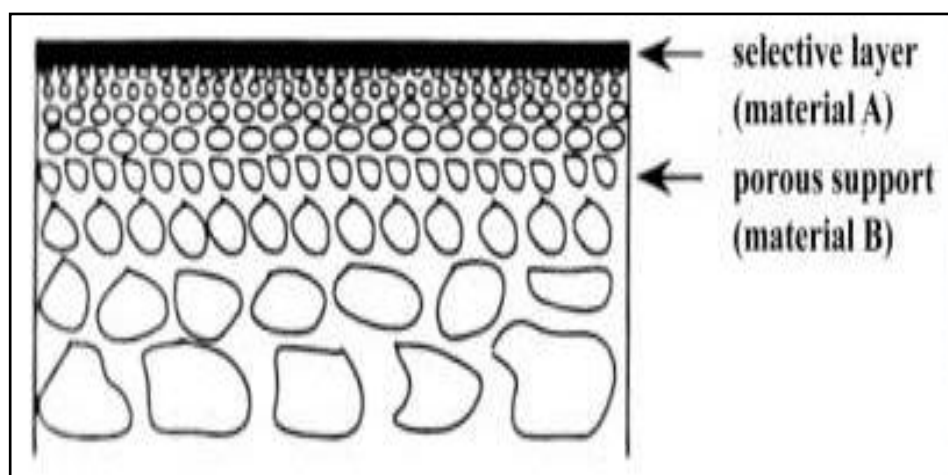
Currently, poly(dimethyl siloxane) (PDMS) is the primary polymer used for olefin separations. In the mid-1950s, PDMS had been found to have a much higher permeability to gases than almost all other synthetic polymers known at that time, and even nowadays PDMS is still considered to be one of the most permeable polymers. This is shown by the research of Shi Y. *et al.* (2006), in their research, poly(dimethyl siloxane) (PDMS) thin film composite membranes originally developed for pervaporation applications for the separation of propylene from nitrogen, and the feasibility of adapting such membranes to olefin recovery from polyolefin purge gas. From their research, the PDMS composite membrane can be adapted to propylene separation from nitrogen that is relevant to propylene recovery from polypropylene degassing off gas.

### 2.5.4 Thin Film Composite Membrane (TFC)

Thin film composite membrane (TFC) is actually the combination of more than one polymer. TFC membrane has been widely applied in gas separation. It is normally fabricated by filling a high permeability and low selectivity polymer into the defects of a highly selective glass polymer asymmetric membrane. The structure of composite membrane can be divided into three layers based on the configuration of the membrane; they are coating layer, selective layer, and porous support layer. The coating layer serves to plug defects in the selective layer and reduce the risk of gas permeation through the defects. The performance and applicability of composite membrane largely depend on the defects filled by the coating material during the fabrication processes, (Zhang *et al.*, 2010).

Composite materials combine the desirable properties of multiple materials. In this sense, composite filters are created by sequential synthesis rather than simply mixing different chemicals. While a variety of approaches are used in their manufacture, most common is the controlled deposition of a material onto a preformed sheet or grid of support material. Composite materials are used when the physical features of one material (for example, the strength of the support material) need to be combined with the chemical features of another (for example, the high charge of the filter material).

Zhang et al., (2010) have been produce composite membrane by coating a thin layer of polyfluoropropylmethylsiloxane (PTFPMS) on the dense layer of asymmetric polyetherimide (PEI) membranes to plug the defects in order to separate  $O_2/N_2$  and  $H_2/N_2$ . Figure 2.5 show the thin film composite membrane with selective layer and porous support. Whereas in **Figure 2.8** shows the thin film composite membrane for RO membrane. **Figure 2.9** show the usual thickness of each layer. While **figure 2.10** shows the usual thickness of each layer (Nunes, 2006).



**Figure 2.8** Thin Film Composite Membrane